IS 7223 (1986): Potassium chloride, analytical reagent [CHD 1: Inorganic Chemicals]

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IS : 7223 - 1986

Indian Standard
SPECIFICATION FOR POTASSIUM CHLORIDE, ANALYTICAL REAGENT
(*First Revision*)

UDC 661.832.321 : 54-4

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6 ***November*** **1986**

AMENDMENT NO. 1 FEBRUARY 1997
TO
IS 7223 : 1986 SPECIFICATION FOR POTASSIUM CHLORIDE, ANALYTICAL REAGENT
(First Revision)

(*Page* 12, *clause* **A-6.3**) — Substitute the following for the existing entries:

'A-6.3 Calculation

Calcium group and magnesium (as Ca), percent by mass (on dry basis) = $\frac{A\,V_1}{2\,M}$

where

A = calcium equivalent in mg of 1 ml of EDTA solution determined in **A-6.2.2,**

V_1 = volume in ml of standard EDTA solution used in **A-6.2.3**, and

M = mass in g of the sample taken for test.'

(*Page* 17, *clause* **A-12.2**) — Substitute the following for the existing clause:

'A-12.2 Preparation of Test Solution

Take 40 ml of the sample solution (*see* **A-2.1.1**), add 15 ml of sodium hydroxide solution and dilute with water to 100 ml. Add 2 ml of Nessler solution in a control test, having an equal volume of the solution containing 2 ml of standard ammonium solution and 1.5 ml of sodium hydroxide solution. Dilute to 100 ml with water, add 2 ml of Nessler solution.'

(*Page* 17, *clause* **A-12.3**) — Delete.

(*Page* 17, *clause* **A-12.4**) — Renumber clause **'A-12.4'** as **'A-12.3'**.

(CHD 002)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR POTASSIUM CHLORIDE, ANALYTICAL REAGENT

(*First Revision*)

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Indian Standard

SPECIFICATION FOR POTASSIUM CHLORIDE, ANALYTICAL REAGENT

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 In this revision, a new requirement for *p*H has been included and requirements for free acid and free alkali have been deleted. Further the requirements for nitrate, phosphate, ammonium salts and arsenic have been modified so as to be in line with the requirements for sodium chloride, analytical reagent grade in IS : 4408-1979*.

0.3 Potassium chloride finds use as an analytical reagent. It is also used for photographic purposes. Technical grade of this material has been covered in IS : 4150-1984†.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960‡. The number of significant placed retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for potassium chloride, analytical reagent.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of small white crystals or crystalline powder free from impurities.

*Specification for sodium chloride, analytical reagent (*first revision*).
†Specification for potassium chloride, technical (*second revision*).
‡Rules for rounding off numerical values (*revised*).

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR POTASSIUM CHLORIDE, ANALYTICAL REAGENT

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Matter insoluble in water, percent by mass, *Max*	0.003	A-3
ii)	Potassium chloride (at KCl), percent by mass, *Min*	99.8	A-4
iii)	Sodium (at NaCl), percent by mass, *Max*	0.05	A-5
iv)	Calcium group and magnesium (as Ca), percent by mass, *Max*	0.004	A-6
v)	Sulphate (as SO_4), percent by mass, *Max*	0.001	A-7
vi)	Iron (as Fe), ppm, *Max*	3	A-8
vii)	Bromide and iodide (as Br), ppm, *Max*	50	A-9
viii)	Nitrate (as NO_2), ppm, *Max*	5	A-10
ix)	Phosphate (as PO_4), ppm, *Max*	5	A-11
x)	Ammonium salts (as NH_4), ppm, *Max*	5	A-12
xi)	Arsenic (as As), ppm, *Max*	0.5	A-13
xii)	Barium (as Ba), ppm, *Max*	10	A-14
xiii)	Heavy metals (as Pb), ppm, *Max*	5	A-15
xiv)	Loss of mass at 150°C, percent by mass, *Max*	0.1	A-16
xv)	*p*H of 5 percent solution	5-9	A-17

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in air-tight bottles with capacity of 500 g, 1 kg and 2.5 kg.

3.2 Marking — The packages shall be securely closed and marked legibly and indelibly with the following information:

a) Name of the material;
b) Mass of the material in the package;
c) Name of the manufacturer and recognised trade-mark, if any; and
d) Lot or batch number, in code or otherwise.

3.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST OF POTASSIUM CHLORIDE, ANALYTICAL REAGENT

A-1. QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF SAMPLE

A-2.1 Sample for All Tests — Crush 100 g of the sample so that it passes through 1.00 mm IS Sieve, and keep it in a clean glass-stoppered weighing bottle in a desiccator for subsequent tests.

A-2.1.1 *Preparation of Solution* — Weigh accurately 50 g of the sample (*see* **A-2.1**), and determine matter insoluble in water as in **A-3**. Make up the filtrate to 500 ml with water. The solution shall be used for the subsequent tests.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Dissolve 50 g of the sample (*see* **A-2.1**), accurately weighed, in about 100 ml of water. Filter it through a tared filter paper

*Specification for water for general laboratory use (*second revision*).

or sintered glass crucible (G No. 4) and wash the residue with water till the filtrate gives no opalescence with silver nitrate solution. Dry the filter paper or sintered glass crucible with the residue to constant mass at 105 to 110°C.

A-3.2 Calculation

Matter insoluble in water, percent by mass = $\frac{M_1}{M_2} \times 100$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of sample taken for the test.

A-4. DETERMINATION OF POTASSIUM

A-4.1 Gravimetric Method Using Tetraphenyl Boron (Referee Method)

A-4.1.1 *Reagents*

A-4.1.1.1 *Concentrated hydrochloric acid — See* IS : 265-1976*.

A-4.1.1.2 *Sodium tetraphenyl boron solution* — Dissolve 1 g sodium tetraphenyl boron in 100 ml of sodium hydroxide solution (0.01 N). Before use, cool the solution in ice.

A-4.1.1.3 *Wash solution* — Recrystallize potassium tetraphenyl boron from acetone and saturate the water with the recrystallized product. The solution shall be prepared fresh.

A-4.1.2 *Procedure* — Weigh 1.00 g of the sample (*see* **A-2.1**), dissolve in water and make up the volume to 100 ml in a measuring flask. Transfer 10 ml of the solution into a beaker, dilute to 50 ml and add 3 ml of concentrated hydrochloric acid. Place the beaker in an ice-bath for 5 to 10 minutes. When the solution has cooled, add 50 ml of sodium tetraphenyl boron solution cooled previously in ice. Mix by gentle swirling and leave in the ice-bath for 5 to 10 minutes. Filter through a sintered glass crucible (G No. 4), or a Gooch crucible, wash the beaker and the precipitate with three 10 ml portions of the wash solution and dry the crucible containing the precipitate for one hour at 110 to 120°C. Cool in a desiccator and weigh.

*Specification for hydrochloric acid (*second revision*).

A-4.1.3 *Calculation*

Potassium chloride (as KCl), percent by mass (on ignition basis) $= 208.0 \frac{M_1}{M_2}$

where

M_1 = mass in g of the precipate, and

M_2 = mass in g of the ignited sample (**A-2.1**) taken for the test.

A-4.2 By Flame Photometer

A-4.2.1 *Simple Flame Photometer* — equipped with interference filter.

A-4.2.2 *Reagents*

A-4.2.2.1 *Standard potassium solution* — Weigh exactly 1 g of potassium chloride, dissolve it in water and dilute to one litre with water in a measuring flask. This solution contains 0.1 g of potassium chloride per 100 ml.

A-4.2.2.2 *Calibration graph* — Take 10, 20, 40, 60 and 80 ml of standard potassium solution (*see* **A-4.2.2.1**) and dilute to 100 ml with water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g of potassium chloride per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in **A-4.2.3** and plot the concentration against galvanometer reading in a rectangular coordination graph. Draw a smooth curve over the points which give a calibration graph in the range 0.01 to 0.1 percent potassium chloride.

A-4.2.2.3 *Sample solution* — Dissolve 1.0 g of the sample (*see* **A-2.1**) in minimum quantity of water and dilute to 1 000 ml in a measuring flask with water.

A-4.2.3 *Procedure*

A-4.2.3.1 Use a flame photometer equipped with atomizer, burner; optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-4.2.3.2 Insert the potassium filter corresponding to wavelength 767 nm. Light the burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 to 0.7 kg/cm^2 and maintain the above air pressure constant such that the flame is nonluminous by turning the gas control knob. First spray water and adjust the pointer to zero in galvanometer scale by zero adjustment

knob. Then spray the potassium chloride standard solution (**A-4.2.1.1**) and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray water to see pointer comes to zero; then spray standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with the same adjustment during both the operations. Now reading zero by water and with the same adjustment 100 by standard solution indicate that the instrument has been now ready for measurement.

A-4.2.3.3 Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared in **A-4.2.2** and obtain a calibration graph in the range 0.01 to 0.1 percent potassium chloride. After each reading of the diluted solutions, spray with water to obtain a zero galvanometer reading. After washing with water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of potassium chloride in the solution (say *A*).

A-4.2.4 *Calculation*

$$\text{Potassium chloride, percent by mass} = 100 \frac{A}{M}$$

where

A = concentration of potassium chloride in the sample solution,

M = mass of the material (*see* **A-2.1**) taken for preparing the sample solution.

A-4.3 Perchloric Method

A-4.3.0 *Principle of Method* — After precipitation of sulphate as barium sulphate, potassium is precipitated but as perchlorate and the precipitate weighed.

A-4.3.1 *Reagents*

A-4.3.1.1 *Concentrated hydrochloric acid* — *See* IS : 265-1976*.

A-4.3.1.2 *Barium chloride solution* — 5 percent.

A-4.3.1.3 *Perchloric acid* — 60 percent.

A-4.3.1.4 *Ethanol* — 96 percent.

A-4.3.2 *Procedure*

A-4.3.2.1 Weigh accurately about 10 g of the sample and transfer it to a 500 ml beaker. Add to the beaker 350 ml of water and 20 ml of concentrated hydrochloric acid and boil for at least half an hour. Add water to the beaker to maintain the total volume of the contents. Add hot

*Specification for hydrochloric acid (*second revision*).

barium chloride solution drop by drop to the boiling solution. Keep the solution boiling and ensure complete precipitation of sulphates by adding barium chloride solution in slight excess. Digest the solution on a water-bath for about half an hour and allow to cool. Transfer the entire contents to a 500-ml volumetric flask, washing the beaker a few times with water. Dilute the contents to the mark.

A-4.3.2.2 Filter a part of the solution in the volumetric flask through a dry filter paper into a dry conical flask. Reject the first 30 ml of the filtrate. Take with a pipette 25 ml of the filtrate into a porcelain dish of 10 cm diameter and add into it 10 ml of perchloric acid. Evaporate the solution on a boiling water-bath and keep stirring the contents of the dish during evaporation. Continue evaporation until thick white fumes of perchloric acid are evolved. Grind the residual mass with a glass rod with 5 ml of perchloric acid and evaporate again until most of perchloric acid has been removed. The residue should not be completely dry, it should still have a syrupy consistency.

A-4.3.2.3 Cool the residue in the dish and add immediately 10 ml of ethanol and stir the mass thoroughly with a glass rod. Allow the residue to settle and decant off the alcohol through a weighed sintered glass crucible (G. No. 4). Add 20 ml ethanol again to the residue, grind the mass with a glass rod and decant off the alochol through the crucible. Finally, transfer the residue to the crucible with ethanol to which 0.2 percent by volume of perchloric acid has been added and wash with the same ethanol. Finally, wash with 4 to 5 ml of ethanol alone. The whole filtrate should be about 75 ml. Dry the precipitate for at least 30 minutes at 130 ± 2°C and cool in a desiccator and weigh.

A-4.3.3 *Calculation*

$$\text{Potassium chloride content (as KCl), percent by mass} = \frac{1\,076{\cdot}2\,M_1}{M}$$

where

M_1 = mass in g of the precipitate, and

M — mass in g of the sample taken for the test in **A-4.3.2.2.**

A-5. DETERMINATION OF SODIUM

A-5.1 Simple Flame Photometer — equipped with interference filter.

A-5.2 Reagents

A-5.2.1 *Standard Sodium Chloride Solution* — Weigh accurately 5 g of sodium chloride A.R., dissolve it in water and transfer to one-litre flask and make up the volume. The standard solution is 0.5 g sodium chloride per 100 ml.

A-5.2.2 *Standard Solution of KCl for Buffer* — Recrystallize A.R. or G.R. potassium chloride and dry at 110°C. Dissolve 10 g of the recrystallized potassium chloride in 100 ml water.

A-5.2.3 *Sample Solution* — Weigh 1 g of the sample (*see* **A-2.1**) and dissolve it in 1 000 ml of water.

A-5.2.4 *Calibration Graph* — Since sodium chloride is an impurity in potassium chloride, which may interfere in the determination of sodium chloride, bracketing technique or internal calibration of sodium chloride in presence of potassium chloride is adopted. Buffer standard solution is used for calibration.

A-5.2.5 *Buffer Standard NaCl Solution* — bracketed with potassium chloride.

A-5.3 Procedure

A-5.3.1 Pipette 1, 2, 4, 5, 6, 8 and 10 ml of standard sodium chloride solution prepared in **A-5.2.1** into 100-ml measuring flasks. Each flask contains 5, 10, 20, 25, 30, 40, 50 mg sodium chloride. Pipette 10 ml of potassium chloride (1.0 g.KCl) standard solution prepared in **A-5.2.2** to all the flasks containing sodium chloride standard solution (5 to 50 mg NaCl). Make up the volume to 100 ml. This is buffered solution with potassium chloride. Use this solution for calibration graph as described earlier for potassium chloride (*see* **A-4.2.3**) using sodium filter 589 nm instead of potassium filter. Use the buffer solution containing 0.05 g of sodium chloride per 100 ml for obtaining maximum deflection of 100 division of galvanometer. Sodium chloride range in the calibration graph is 0.005 to 0.05 g sodium chloride per 100 ml and sodium chloride as impurity exists in the same proportion as potassium chloride.

A-5.3.2 After washing with water, spray the sample solution (*see* **A-5.2.3**) containing approximately 0.1 g of potassium chloride per 100 ml and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium chloride per 100 ml, say *A*.

A-5.4 Calculation

$$\text{Sodium chloride, percent by mass} = \frac{A}{M} \times 100$$

where

A = concentration of sodium chloride in g per 100 ml in the sample solution, and

M = mass of sample taken for preparing the sample solution in g per 100 ml.

A-6. DETERMINATION OF MAGNESIUM AND CALCIUM

A-6.1 Reagents

A-6.1.1 *Standard Calcium Solution* — Weigh 1.00 g of calcium carbonate dried at 120°C and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to one litre in a graduated flask. One millilitre of the solution is equivalent to 0.400 8 mg of calcium (as Ca).

A-6.1.2 *Standard (EDTA) Solution* — Dissolve 3.72 g of disodium ethylene diamine tetraacetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against the standard calcium solution, following the procedure given in **A-6.2.**

A-6.1.3 *Eriochrome Black T Indicator Solution* — Dissolve 0.1 g of the dye in 20 ml of rectified spirit conforming to IS : 323-1959*. This solution shall be prepared fresh every week.

A-6.1.4 *Ammonium Chloride-Ammonium Hydroxide Buffer Solution* — Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetraacetate dihydrate and 0.616 g of magnesium sulphate ($MgSO_4.7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to one litre.

NOTE — Five millilitres of the buffer solution added to 50 ml water should not consume more than a drop of EDTA solution to change its colour to distinct blue with eriochrome black T indicator.

A-6.2 Procedure

A-6.2.1 *Standardization of EDTA Solution* — Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against the standard EDTA solution to a pure blue end point.

A-6.2.2 Titrate 25 ml of the buffer solution against EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml), from the reading obtained in **A-6.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml EDTA solution (say *A*).

A-6.2.3 Transfer exactly 100 ml of the sample solution (*see* **A-2.1.1**) into a 250-ml conical flask. Add 20 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine-red colour of the solution changes to pure blue end-point. Note the volume of EDTA solution used in the titration.

*Specification for rectified spirit (*revised*).

A-6.3 Calculation

$$\text{Calcium group and magnesium (as Ca), percent by mass (on dry basis)} = \frac{A\,V_1}{M}$$

where

A = calcium equivalent in mg of 1 ml of EDTA solution determined in **A-6.2.2.**

V_1 = volume in ml of standard EDTA solution used in **A-6.2.3,** and

M = mass in g of the sample taken for the test.

A-7. DETERMINATION OF SULPHATE

A-7.1 Method A (Gravimetric Method)

A-7.1.1 *Reagents*

A-7.1.1.1 *Dilute hydrochloric acid* — approximately 4 N.

A-7.1.1.2 *Barium chloride solution* — approximately 10 percent.

A-7.1.2 *Procedure* — Transfer exactly 100 ml of the solution (*see* **A-2.1.1**) in about 400 ml of water, filter and wash the residue to free it from soluble salts. Collect the filtrate and wash. Add one drop methyl orange and 10 ml or more of dilute hydrochloric acid till it is pink and then boil. Add to the boiling solution, 10 to 12 ml barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 minutes to obtain a granular precipitate. Allow to stand for 4 hours and filter through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash the precipitate till free of chloride, and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and determine as barium sulphate.

A-7.1.3 *Calculation*

$$\text{Sulphate (as } SO_4\text{), percent by mass} = \frac{M_1 \times 41{\cdot}13}{M}$$

where

M_1 = mass in g of barium sulphate precipitate, and

M = mass in g of the sample taken for the test.

A-7.2 Method B (Turbidimetric Method)

A-7.2.1 *Apparatus*

A-7.2.1.1 *Barium chloride scoop* — for dispensing 0.20 ± 0.02 g of barium chloride.

A-7.2.1.2 *Magnetic stirrer* — with small-sized plastic covered stirring bar.

A-7.2.1.3 *Colorimetric equipment* — A spectrophotometer or filter photometer, equipped with cells of 5-cm light path.

A-7.2.2 *Reagents*

A-7.2.2.1 *Barium chloride* — crystalline, of crystal size 50 to 500 microns.

A-7.2.2.2 *Glycerine-alcohol solution* — Add 1 volume of glycerine to 2 volumes of 95-percent rectified spirit, and mix thoroughly.

A-7.2.2.3 *Sodium chloride-hydrochloric acid solution* — Add 67 g of sodium chloride and 8 ml concentrated hydrochloric acid to 200 ml of water, and shake intermittently to dissolve the sodium chloride. It is not necessary to filter off the excess sodium chloride.

A-7.2.2.4 *Standard sulphate solution* — Dissolve 0.133 2 g, weighed to 0.1 mg, of dried anhydrous sodium sulphate in water, and dilute to volume in a one-litre volumetric flask. Mix thoroughly. One millilitre of the solution contains 0.090 mg of sulphate (as SO_4).

A-7.2.3 *Preparation of Calibration Curve*

A-7.2.3.1 Prepare a blank by introducing from a burette 5 ml of water to a clean, dry 100-ml beaker and from a pipette, 10 ml of glycerine-alcohol solution, add 5 ml of sodium chloride-hydrochloric acid solution. Insert the stirring bar in the beaker, place the beaker on the magnetic stirrer, and stir at a moderate rate. Simultaneously, start a stop watch and add one scoopful of barium chloride to the solution in the beaker. Stop the stirrer after 60 seconds, and transfer the solution to the dry colorimeter cell. Place the cell in position in the colorimeter.

A-7.2.3.2 Prepare two each of the following four calibration standards, carrying each to completion before beginning the succeeding one:

Sulphate (SO_4)	*Standard Sulphate Solution*	*Water*
mg	ml	ml
0.45	5.0	45
0.90	10.0	40
1.35	15.0	35
1.80	20.0	30

Add from a pipette 100 ml of glycerine-alcohol solution and 5 ml of sodium chloride-hydrochloric acid solution. Add one scoopful of barium chloride and develop the turbidity as directed for the blank. Measure

the absorption of each standard versus the blank at 425 nm within 3 to 6 minutes after starting the stop watch. Plot the average absorbance values for each of the four standards against the corresponding concentration of sulphate. Periodically check the 0.90 mg standard. This is especially important for an analyst without previous experience of the procedure.

A-7.2.4 *Procedure* — Select an aliquot of the sample that contains between 0.45 and 1.8 mg of sulphate (as SO_4). If the sample contains less than 0.45 mg of sulphate, add from a pipette 10 ml standard sulphate solution to the sample or aliquot. Add water to make the volume to 50 ml. Develop the turbidity and measure the absorbance as in **A-7.2.3.** Refer to the calibration curve to obtain the quantity of sulphate in the sample or aliquot. Correct the gross amount found for the amount of sulphate in the standard sulphate solution, if added.

A-8. TEST FOR IRON

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinder* — 100 ml capacity.

A-8.2 Reagents

A-8.2.1 *Thioglycolic Acid* — containing not less than 97 percent thioglycolic acid by mass.

A-8.2.2 *Concentrated Ammonium Hydroxide* — relative density 0.90.

A-8.2.3 *Standard Iron Solution* — Dissolve 0.702 g of ammonium ferrous sulphate in about 100 ml water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-8.3 Procedure

A-8.3.1 Take exactly 50 ml of the sample (*see* **A-2.1.1**) and transfer the solution to a Nessler cylinder. Dilute to 80 ml with water and add 4 drops of thioglycolic acid followed by 1 ml of concentrated ammonium hydroxide.

A-8.3.1.1 Carry out a control test in another Nessler cylinder using 1.5 ml dilute standard iron solution and the same quantities of the other reagents. Dilute the solution in both the cylinders to 100 ml and mix well.

A-8.3.1.2 The requirement specified in Table 1 shall be taken as not having exceeded, if the intensity of colour produced with the material is not greater than that obtained in the control test.

A-9. DETERMINATION OF BROMIDE AND IODIDE

A-9.1 Reagents

A-9.1.1 *Anhydrous Disodium Hydrogen Orthophosphate*

A-9.1.2 *Sodium Hypochlorite Solution* — containing 2 percent available chlorine.

A-9.1.3 *Sodium Formate*

A-9.1.4 *Potassium Iodide* — solid.

A-9.1.5 *Dilute Sulphuric Acid* — approximately 2 N.

A-9.1.6 *Ammonium Molyhdate Solution* — 10 percent (*m/v*).

A-9.1.7 *Standard Sodium Thiosulphate Solution* – 0.02 N.

A-9.1.8 *Starch Indicator Solution* — Titrate 5 g of starch and 0.01 g of mercuric iodide against 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

A-9.2 Procedure — Dissolve 15 g sample (*see* **A-2.1**) and 1 g of anhydrous disodium hydrogen orthophosphate in 45 ml of water and add 5 ml of sodium hypochlorite solution (free from bromide and iodine) containing 2 percent of available chlorine. Heat to boiling for 1 minute, add solution of 2.5 g of sodium formate in 5 ml of water, boil again for 5 minutes, cool and dilute to 100 ml with water. Add 1 g of potassium iodide, 30 ml of dilute sulphuric acid and 1 drop of ammonium molybdate solution. Titrate it against standard sodium thiosulphate solution using starch solution as indicator.

A-9.2.1 The limits prescribed in Table 1 shall be taken as not having exceeded if not more than 2.8 ml of standard sodium thiosulphate solution is required.

A-10. TEST FOR NITRATES

A-10.1 Reagents

A-10.1.1 *Dilute Sulphuric Acid* — approximately 5 N.

A-10.1.2 *Concentrated Hydrochloric Acid — See* IS : 265-1976*.

A-10.1.3 *Standard Potassium Nitrate Solution* — 0.001 N.

A-10.1.4 *Concentrated Sulphuric Acid – See* IS : 266-1977†.

*Specification for hydrochloric acid (*second revision*).
†Specification for sulphuric acid (*second revision*).

A-10.1.5 *Standard Indigo Carmine Solution* — Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid, add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to make up the volume to 1 000 ml. Standardize the solution so that 100 ml added to 3.3 ml potassium nitrate solution is just decolourized on adding 13 ml concentrated sulphuric acid and heating to boiling. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.002 mg of nitrate (as NO_3).

A-10.1.6 *Dilute Ammonium Hydroxide Solution* — approximately 5 N.

A-10.2 *Procedure* — Dilute accurately 20 ml of the sample (*see* **A-2.1.1**) with 100 ml of water. Add 50 ml dilute ammonium hydroxide solution and filter by suction. To 15 ml of the filtrate, add 5 ml of dilute sulphuric acid, 5 ml of standard indigo carmine solution and 20 ml of concentrated sulphuric acid, and heat to boiling.

A-10.2.1 The limits prescribed in Table 1 shall be taken as not having exceeded if the blue colour produced does not enitrely disappear.

A-11. TEST FOR PHOSPHATES

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 *Dilute Sulphuric Acid* — approximately 5 N and 1 N.

A-11.2.2 *Phosphate Reagent A* — Dissolve without heating 5 g of powdered ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

A-11.2.3 *Phosphate Reagent B* — Dissolve without heating 0.2 g of methyl-*ρ*-aminophenol sulphate (metol), and 20 g of sodium metabisulphite in 100 ml of water.

A-11.2.4 *Standard Phosphate Solution* — Dissolve 0.286 0 g of potassium dihydrogen phosphate (KH_2PO_4) in water and make up the volume to 1 000 ml. Dilute 10 ml of this solution to 100 ml with water. One millilitre of the solution is equivalent to 0.02 mg of phosphate (as PO_4). Keep the solution in a waxed bottle.

A-11.3 Procedure — Take 20 ml of the sample (*see* **A-2.1.1**) in a Nessler cylinder, add 7 ml of dilute sulphuric acid (5 N) and 1 ml of each of the phosphate reagents A and B. Dilute with water to 50 ml and place in a water-bath at 60°C for 10 minutes. Carry out a control test using 1 ml of standard phosphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-11.3.1 The limits prescribed in Table 1 shall be taken as not having exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-12. TEST FOR AMMONIUM SALTS

A-12.0 Outline of the Method — The solution of the sample is treated with sodium hydroxide and filtered. To the filtrate, Nessler solution is added and the colour produced is compared with that produced in the control test containing definite amount of ammonium hydroxide.

A-12.1 Reagents

A-12.1.1 *Sodium Hydroxide Solution* — 10 percent (*m/v*).

A-12.1.2 *Nessler Solution* — Dissolve 143 g of sodium hydroxide in 700 ml of water Dissolve 50 g of red mercuric iodide and 40 g of potassium iodide in 200 ml of water. Pour the iodide solution into hydroxide solution and dilute to 1 000 ml. Allow to settle and use the clear supernatant liquid.

A-12.1.3 *Standard Ammonium Chloride Solution* — Dissolve 0 296 g of ammonium chloride (NH_4Cl) in water and dilute to 100 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of ammonium (as NH_4).

A-12.2 Preparation of Test Solution – Dilute 25 ml of the sample solution (*see* **A-2.1.1**) to about 200 ml of water, filter carefully, if necessary, and dilute to 250 ml in a measuring flask (solution A).

A-12.3 Dilute 10 ml of the solution A (**A-12.2**) to 45 ml and add 15 ml of sodium hydroxide solution. Filter through a sintered glass crucible previously washed with sodium hydroxide solution. Dilute with water to 100 ml and add 2 ml of Nessler solution. In a control test, having an equal volume of the solution and containing 2 ml of standard ammonium chloride solution and 1.5 ml of sodium hydroxide, add 2 ml of Nessler solution.

A-12.4 The limits prescribed in Table 1 shall be taken as not having exceeded if the colour produced with the material is not darker than that produced with the standard solution.

A-13. TEST FOR ARSENIC

A-13.1 Procedure — Take 25 ml of the prepared sample (*see* **A-2.1.1**) and add 10 ml of water and carry out test for arsenic as prescribed in IS : 2088-1983* using 0 001 32 mg of arsenic trioxide (As_2O_3) for preparing the comparison stain.

*Methods for determination of arsenic (*second revision*).

A-14. TEST FOR BARIUM

A-14.1 Apparatus

A-14.1.1 *Nessler Cylinders* — 100-ml capacity.

A-14.2 Reagents

A-14.2.1 *Dilute Hydrochloric Acid* — 1 N.

A-14.2.2 *Barium Chloride Solution* — approximately 10 percent (*m/v*).

A-14.2.3 *Dilute Sulphuric Acid* — 5 N.

A-14.2.4 *Rectified Spirit — See* IS : 323-1959*.

A-14.2.5 *Standard Sulphate Solution* — Dissolve 0.148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of sulphate (as SO_4).

A-14.3 Procedure — Dissolve 50 ml of the sample (*see* **A-2.1.1**) in 40 ml of water and add 10 ml of rectified spirit, mix, add 0.5 ml of dilute sulphuric acid, mix immediately and set aside for 1 hour. To another Nessler cylinder add 0.5 ml of standard sulphate solution, 37 ml of water and 10 ml of rectified spirit and 1 ml of dilute hydrochloric acid, mix and add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 hour.

A-14.3.1 The limits prescribed in Table 1 shall be taken as not having exceeded if the turbidity produced with the material is not greater than that produced with the standard solution.

A-15. TEST FOR HEAVY METALS

A-15.1 Apparatus

A-15.1.1 *Nessler Cylinders* — 50-ml capacity.

A-15.2 Reagents

A-15.2.1 *Acetic Acid* — approximately 33 percent (*v/v).*

A-15.2.2 *Dilute Ammonium Hydroxide* — approximately 4 N.

A-15.2.3 *Potassium Cyanide Solution* — Dissolve 10 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide (20 volume strength), allow to stand for 24 hours and make up to 100 ml with water.'

A-15.2.4 *Sodium Sulphide Solution* — Dissolve 10 g of sodium sulphide ($Na_2S.9H_2O$) in 100 ml of water.

*Specification for rectified spirit (*revised*).

A-15.2.5 *Standard Lead Solution* — Dissolve 0.160 g of lead nitrate in 5 ml of concentrated nitric acid (*see* IS : 264-1976*) and dilute to 1 000 ml in a graduated flask. Again dilute 100 ml of the solution to 1 000 ml. One millilitre of the solution finally obtained contains 0.01 mg of lead (as Pb).

A-15.3 Procedure — Dilute 25 ml solution (**A-2.1.1**) in water in a Nessler cylinder and add 5 ml of acetic acid. Make the mixture alkaline with dilute ammonium hydroxide and add 1 ml of potassium cyanide solution, if turbid, filter. Add two drops of sodium sulphide solution and mix well. Carry out a control test in another Nessler cylinders in exactly the same manner but using 1 ml of standard lead solution. Dilute the solution in both the cylinders to 50 ml mark. Compare the colour produced in the two cylinders against a white background.

A-15.3.1 The material shall be taken to have not exceeded the limit specified in Table 1 if the intensity of colour obtained with the material is not greater than that obtained in the control test.

A-16. DETERMINATION OF LOSS OF MASS AT 150°C

A-16.1 Apparatus

A-16.1.1 *Weighing Bottle* — approximately 100 ml capacity and 75 mm in diameter, with ground-glass stopper.

A-16.1.2 *Electric Oven* — Capable of reaching a temperature of at least 150°C, regulated so as not to exceed a temperature of 170°C.

A-16.2 Procedure — Weigh 5 g of the material to the nearest 0.1 mg. Spread thin layers of the material in the weighing bottle previously dried for 30 minutes in the oven controlled at a temperature of 150°C and allowed to cool to room temperature in a desiccator and weigh. Place it in the oven with the ground-glass stopper of the weighing bottle slant-wise. Gradually heat in the oven from the starting temperature (which should not exceed 100°C) to at least 150 ± 2°C and maintain this temperature for 1 hour. Remove the weighing bottle, place it in a desiccator to cool and after cooling to room temperature, weigh it again with stopper in position.

A-16.3 Calculation

$$\text{Loss on heating at 150°C, percent by mass} = \frac{(M_1 - M_2) \times 100}{M}$$

where

M_1 = mass in g of the material and weighing bottle before heating,

M_2 = mass in g of the material and weighing bottle after heating, and

M = mass in g of the material taken for the test.

*Specification for nitric acid (*second revision*).

A-17. DETERMINATION OF *p*H VALUE

A-17.1 Procedure — Weigh 10 g sample and transfer into 250 ml conical flask. Add 200 ml water and put rubber stopper. Shake on wrist action shaker or any similar device for one hour. Transfer the contents into a 250-ml beaker and allow to settle for about 30 minutes. Measure *p*H value of supernatant solution using *p*H meter, (slight black turbidity in supernatant solution does not materially affect the results). The *p*H meter should have been previously calibrated using standardized buffer solutions.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF POTASSIUM CHLORIDE, ANALYTICAL REAGENT

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 The samples shall be placed in suitable, clean, dry containers.

B-1.3 The samples containers shall be of such size that they are almost completely filled by the sample.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full details of sampling and the date of sampling.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested for each lotfor ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number *(n)* of containers to be selected from the lot shall depend on the size of the lot (*N*) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING
(*Clause* B-2.2)

LOT SIZE	No. OF CONTAINERS TO BE SELECTED
N	*n*
(1)	(2)
4 to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 These containers shall be selected at random from the lot, and in order to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any one count them 1, 2, 3, ... , etc, up to *r* and so on, where *r* is the integral part of *N/n*. Every *r*th container thus counted shall be taken out.

B-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 For drawing samples, an auger shall be used.

B-3.2 Introduce the auger in different parts of each container to draw an appropriate quantity of the sample from each container.

B-3.3 Out of the portions collected from all the selected containers, an equal quantity of the material shall be taken out and mixed together to form a composite sample of about 250 g. The composite sample so formed shall be divided into 3 equal parts; one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 The remaining portion of the material from each container shall be divided into three equal parts and each such separate part shall constitute an individual sample. One set of individual samples (representing the *n* containers selected) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.5 All the individual and composite samples shall be immediately transferred to separate glass bottles and labelled with full identification particulars on the bottles.

B-3.6 The referee samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for moisture, potassium chloride content and matter insoluble in water shall be conducted on each of the individual samples and tests for all other characteristics given in **2** and Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 A lot shall be considered as conforming to the requirements of this specification if the stipulations laid down in **B-5.1.1** and **B-5.1.2** are fulfilled.

B-5.1.1 *For Potassium Chloride* — From the individual test results for potassium chloride content the *(X)* and the range *(R)* or mean range ($\bar{R}$) shall be calculated. (Range is the difference between the maximum and minimum of test results). When the sample size is 10, the test results shall be re-arranged in two groups of 5 each in the order of their occurrence and the range for each group shall be computed. $\bar{R}$ will be the mean of these two R's. The lot shall be deemed satisfactory for this characteristic if ($\bar{x}$ — 0.6 *R*) or ($\bar{x}$ — 0.6 $\bar{R}$) is greater than the minimum specified in Table 1.

B-5.1.2 *For Other Characteristics* — The test results for all other characteristics prescribed in col 2 of Table 1 shall satisfy the relevant requirements.

(*Continued from page* 2)

Members	*Representing*
SHRI N. V. KAMBLE	Saurashtra Chemicals, Porbandar
SHRI L. K. MALAVIYA (*Alternate*)	
SHRI P. SUBRAMANIAN	Salt Commissioner, Jaipur
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SHRI T. P. VENKATRAMAN	Standard Alkali (Chemicals Division), Bombay

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	ca
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^4

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